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THE LOS ALAMOS SCIENTIFIC LABORATORY APPROACH TO HYDROGEOCHEMICAL AND STREAM SEDIMENT RECONNAISSANCE FOR URANIUM IN THE UNITED STATES

Stephen L. Bolivar

ABSTRACT

The Los Alamos Scientific Laboratory of the United States is conducting a geochemical survey for uranium in the Rocky Mountain states of New Mexico, Colorado, Wyoming, and Montana and in Alaska. This survey is part of a national hydrogeochemical and stream sediment reconnaissance in which four Department of Energy laboratories will study the uranium resources of the United States to provide data for the National Uranium Resource Evaluation program. The reconnaissance will identify areas having higher than background concentrations of uranium in ground waters, surface waters, and water-transported sediments. The reconnaissance data will be combined with data from airborne radiometric surveys and geological and geophysical investigations to provide an improved estimate for the economics and availability of nuclear fuel resources in the United States and to make information available to industry for use in the exploration and development of uranium resources.

The Los Alamos Scientific Laboratory bases its reconnaissance on an extensive literature review on geochemical sampling, the results of pilot studies, and similar sampling programs in Canada, France, Sweden, New Zealand, and the United Kingdom. The reconnaissance consists of standardized field procedures, data acquisition and evaluation, and publication of results. Water and sediment samples are collected at a nominal density of one sample location per 10 km² except for lake areas of Alaska where the density is one sample location per [illegible]3 km². Water samples are analyzed for uranium by fluorometry which has a 0.0[illegible] parts per billion lower limit of detection. Concentrations of 1[illegible] additional elements in water are determined by plasma-source emission spectrography. All sediments are analyzed for uranium by delayed-neutron counting and a [illegible]0 parts per billion lower limit of detection, which is well below the range of uranium concentrations in natural sediment samples. Elemental concentrations in sediments are also determined by neutron activation analysis for [illegible] elements, by x-ray fluorescence for 9 elements, and by arc-source emission spectrography for [illegible] elements. The multielement analyses provide valuable data for studies concerning pathfinder elements, environmental pollution, elemental distributions, dispersion halos, and economic ore deposits other than uranium. An average of [illegible]0 samples are analyzed by each analytical method each working day.

To date, all of four Rocky Mountain states and about 80% of Alaska have been sampled. About [illegible]70 000 samples have been collected from an area of nearly [illegible] 500 000 km². The philosophy, sampling methodology, analytical techniques, and progress of the reconnaissance are described in several published pilot study, reconnaissance, and technical reports. The Los Alamos program was designed to maximize the identification of uranium in terrains of varied geography, geology, and climate and is one of the largest geochemical programs of this type in the world. Due to its diversity, its technology could be applied to any country.

INTRODUCTION

In 1973, the United States Atomic Energy Commission initiated a National Uranium Resource Evaluation (NURE). The NURE program, now administered by the Department of Energy (DOE), consists of hydrogeochemical and stream-sediment reconnaissance, airborne radiometric surveys, and topical geologic studies (US DOE, 1979).

This paper describes the role of the Los Alamos Scientific Laboratory (LASL) in the Hydrogeochemical and Stream Sediment Reconnaissance (HSSR) initiated in 1975 (ERDA, 1975). The objective of the HSSR program is to complete a systematic reconnaissance of the nation's surface waters, ground waters, and stream sediments. In all likelihood, data from the HSSR program will not identify ore bodies, but rather, they will help outline geochemical provinces favorable for detailed follow-up studies. Four DOE laboratories, the Lawrence Livermore Laboratory, Los Alamos Scientific Laboratory, Oak Ridge Gaseous Diffusion Plant, and the Savannah River Laboratory, have conducted the hydrogeochemical program.

The LASL is conducting the HSSR program for the DOE in the Rocky Mountain states of New Mexico, Colorado, Wyoming, Montana, and Alaska, as well as in parts of Arizona, Utah, and Idaho (Fig. 1). Approximately 250 000 samples from more than 170 000 locations within an approximate 2.7 million km² land area will be sampled by the end of this survey (Sharp, 1977). Priority areas are assigned by the DOE each fiscal year. These areas are then sampled by LASL-supervised subcontractors. The LASL analyzes the samples and reports the data according to DOE priorities. Because large numbers of samples are handled during this project, the LASL has developed rapid, cost-effective, and precise computer-automated analytical systems for analyses by neutron activation analysis, delayed neutron counting, arc- or plasma-source emission spectrography, x-ray fluorescence, and fluorometry. In addition to uranium, thorium, and lithium analyses requested by the DOE, the LASL independently provides analyses for 41 additional elements in each HSSR report. To handle the large amount of data generated in this program, a sophisticated data-base management system has been developed, which is capable of storing and statistically manipulating as many as 140 pieces of information for each sample location (Bolivar, 1979).

THE APPROACH TAKEN

Reviews of geochemical exploration for uranium have been compiled by Boyle et al (1971), Bowie et al (1971), Grimbert (1973), Dall'Aglio (1972), Rose (1977), and Sharp and Bolivar (1980), among others. In addition, large-scale geochemical surveys have been conducted in Canada, Finland, France, Norway, United Kingdom, and the USSR. Most procedures and ideas presented in this paper are a direct consequence of similar programs developed in other countries and of standard geochemical sampling practices summarized by Hawkes and Webb (1962) and Levinson (1974).

Initially, a thorough literature research was conducted on the topics of uranium geochemistry, regional geology, climate, structure, known types of ore deposits in the area, proven methods of exploration, and various types of equipment available for field sampling and field measurements. An initial program was set up based on this research, and in addition, a map and document library was formed.

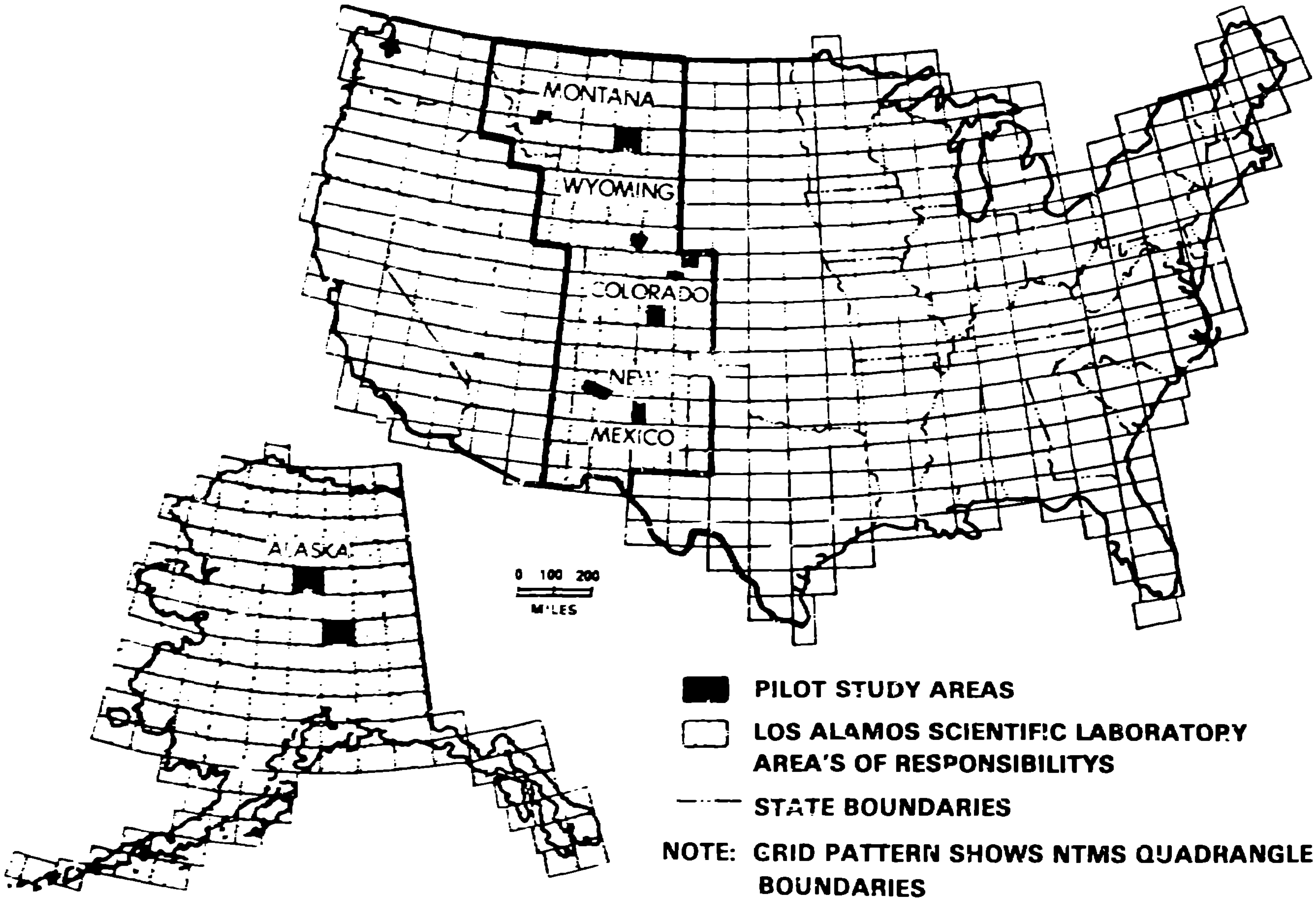


Fig. 1. Index map for the LASL area of responsibility for the HSSR.

Because uranium deposits seldom occur under simple geochemical conditions, to develop a successful HSSR program, a thorough test of all sampling methodologies and philosophies is necessary. Most geochemical anomalies result from the movements of natural waters and soils; therefore, it is also necessary to understand the geometry, size, and type of dispersion patterns that may exist and how they are influenced by geology, climate, and topography (Lovering et al, 1956). This is done by means of pilot (orientation) studies. The ideal place to conduct pilot studies is in the vicinity of known uranium deposits characteristic of the region being studied, where the extent of dispersion halos for anomalies related directly to ore bodies can be determined. The areas should not be contaminated by human activity so that natural geochemical patterns can be observed and compared to background levels in unmineralized terrane (Hawkes and Webb, 1962). However, such areas may not be available or may be limited to small deposits. Studies should cover the full range of environmental and climate conditions typical of the study area (Bolivar, 1979). The pilot studies completed by the LASL are shown in Fig. 1 (Sharp and Aamodt, 1976; Olsen, 1977; Aamodt, 1978; and Sharp et al, 1979).

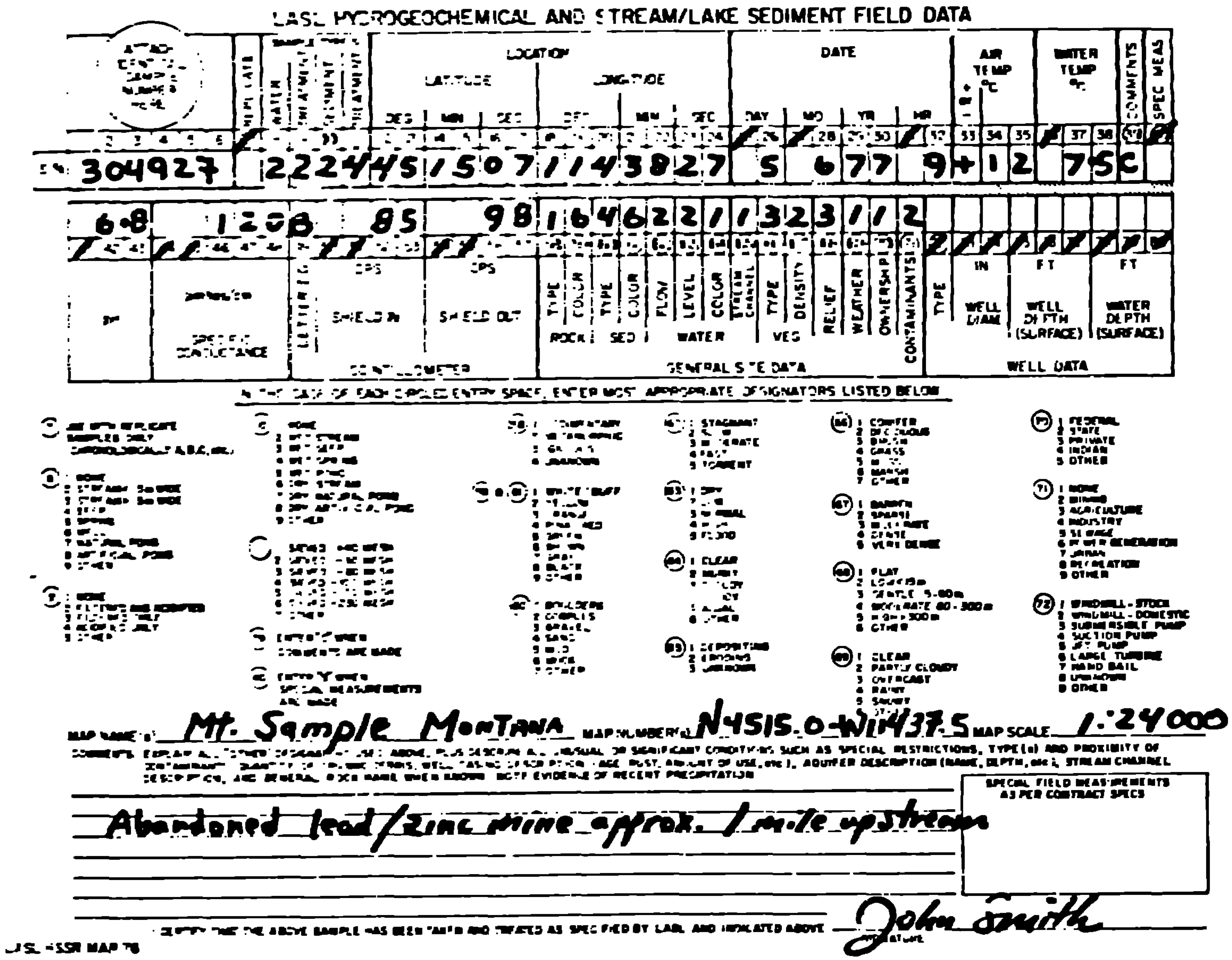
The LASL compiled a field procedures manual that explains the purpose of the program; the care, calibration, and use of field equipment; and the general procedures to be followed for all aspects of the program (Sharp and Aamodt, 1978). Because varying methods of collection and sample preparation affect the sensitivity of geochemical surveys, field procedures and equipment are continuously updated, and pilot studies are conducted for each new region.

Samples are collected by subcontractors according to systematic and standardized sampling procedures as outlined in the sample collection manual of Sharp and Aamodt (1978). All field equipment necessary to collect samples, including sample vials and data forms, and to take and record the required measurements are provided by the LASL. Subcontracted field personnel are required to attend a short training course during which the objectives of the program, sampling methodology, and care and calibration of field equipment are taught. Samplers are required to be able to read a topographic map and recognize geologic regimes. The DOE provides identification cards which are issued by LASL personnel after a prospective sampler attends the training course. In addition, the LASL provides a public relations brochure, written for the layman, explaining the HSSR (Aamodt, 1977). Laboratory personnel are present in a supervisory capacity to monitor the sampling and provide help with equipment malfunctions and obtaining access to private property.

Contract areas generally cover one or more National Topographic Map Series (NTMS) quadrangles; each NTMS quadrangle consists of an area of 1° latitude and 2° or 3° longitude. Access requires four-wheel drive vehicles, which usually are provided by the subcontractor. However, sample collection in some mountainous terrains involves the use of horses or backpacking. Most areas in Alaska are sampled by use of helicopters.

FIELD OBSERVATIONS

In a reconnaissance program, chances are high that any particular sample location will not be revisited. Therefore, it is essential to record all field measurements and observations at the collection site. To do this, the LASL has developed a data form on which the sampler can record the sample type, location, weather, possible contaminants, field measurements, and geologic observations (Fig. 2). Field observations are number coded and eas



LASL HYDROGEOCHEMICAL AND STREAM/LAKE SEDIMENT FIELD DATA

LOCATION — LATITUDE (DEG, MIN, SEC), LONGITUDE (DEG, MIN, SEC); DATE (DAY, MO, YR, HR); AIR TEMP °C; WATER TEMP °C; COMMENTS; SPEC MEAS

304927 2224 45 15 07 114 38 27 5 6 77 9 +12 7.5 C

6.8 1208 85 98 1 6 4 6 2 2 1 1 3 2 3 1 1 2

pH; SPECIFIC CONDUCTANCE; LITHOLOGY; CPS SHIELD IN; CPS SHIELD OUT; SCINTILLOMETER; TYPE, COLOR — ROCK; TYPE, COLOR — SED; FLOW, LEVEL, COLOR, STREAM CHANNEL — WATER; TYPE, DENSITY — VEG; RELIEF; WEATHER; OWNERSHIP; CONTAMINANTS; GENERAL SITE DATA; TYPE; WELL DIAM (IN); WELL DEPTH (SURFACE) (FT); WATER DEPTH (SURFACE) (FT); WELL DATA

IN THE CASE OF EACH CIRCLED ENTRY SPACE, ENTER MOST APPROPRIATE DESIGNATORS LISTED BELOW

(66) 1 CONIFER 2 DECIDUOUS 3 BRUSH 4 GRASS ... 8 MARSH 7 OTHER
(67) 1 BARREN 2 SPARSE 3 MODERATE 4 DENSE 5 VERY DENSE
(68) 1 FLAT 2 LOW <15m 3 GENTLE 5-60m 4 MODERATE 60-300m 5 HIGH >300m 6 OTHER
(69) 1 CLEAR 2 PARTLY CLOUDY 3 OVERCAST 4 RAINY 5 SNOWY
(70) 1 FEDERAL 2 STATE 3 PRIVATE 4 INDIAN 5 OTHER
(71) 1 NONE 2 MINING 3 AGRICULTURE 4 INDUSTRY 5 SEWAGE 6 POWER GENERATION 7 URBAN 8 RECREATION 9 OTHER
(72) 1 WINDMILL - STOCK 2 WINDMILL - DOMESTIC 3 SUBMERSIBLE PUMP 4 SUCTION PUMP 5 JET PUMP 6 LARGE TURBINE 7 HAND BAIL 8 UNKNOWN 9 OTHER

MAP NAME(S) Mt. Sample Montana MAP NUMBER(S) N4515.0-W11437.5 MAP SCALE 1:24000

COMMENTS: EXPLAIN ALL "OTHER" DESIGNATORS USED ABOVE, PLUS DESCRIBE ALL UNUSUAL OR SIGNIFICANT CONDITIONS SUCH AS SPECIAL RESTRICTIONS, TYPE(S) AND PROXIMITY OF CONTAMINANT, ... AQUIFER DESCRIPTION (NAME, DEPTH, etc.), STREAM CHANNEL DESCRIPTION, AND GENERAL ROCK NAME WHEN KNOWN. NOTE EVIDENCE OF RECENT PRECIPITATION.

Abandoned lead/zinc mine approx. 1 mile upstream

SPECIAL FIELD MEASUREMENTS AS PER CONTRACT SPECS

I CERTIFY THAT THE ABOVE SAMPLE HAS BEEN TAKEN AND TREATED AS SPECIFIED BY LASL AND INDICATED ABOVE John Smith SIGNATURE

LASL HSSR MAP 78

Fig. 2. The LASL data form.

be recorded in minimal time. Each form has additional space for comments or clarification of information. This field data form produces four carbon copies and is weather resistant. Coded prenumbered adhesive stickers are attached to each data form and are placed on all samples so that the state and quadrangle from which the sample was taken can be identified easily.

The following observations are generally recorded at each location site.

<u>location</u> - The LASL supplies subcontractors with at least two copies of all field maps. These maps are generally 7.5 minute (1:24 000), 15 minute (1:62 500), NTMS topographic (1:250 000), or planimetric county road maps. Each map contains a sample grid and/or premarked sample location After sample collection, locations are marked on the field map. Later these locations are transferred to an unrolled copy on which latitude and longitude can be calculated.

<u>weather</u> - Seasonal climatic conditions may drastically affect uranium concentrations in surface waters and, to a much lesser extent, in sediments (Fix, 1956; Germanov et al, 1958; Doi et al, 1975; Rose et al, 1976). Consequently, hydrogeochemical surveys should be completed as rapidly as possible. During periods of high runoff, normal uranium concentrations may be diluted, whereas after a prolonged drought, uranium concentrations in runoff may be increased for a short period (Peacock, 1961; Lopatkina, 1964).

<u>relief</u> - Several elements, including uranium, in both surface waters and sediments tend to have relatively short dispersion patterns in areas of high relief (Chamberlain, 1964). Furthermore, access to water may be difficult and sediment may be absent locally. Therefore, sample densities may have to be increased so that adequate coverage can be obtained in areas of high relief.

<u>geology</u> - Uranium content in both water and sediment generally reflects the local geology. For example, because of complexing of uranium with carbonate ions, a stream flowing over carbonate terrane would tend to have a higher uranium content than a similar stream flowing over siliceous terrane (Levinson, 1974). Sediments from acidic igneous rocks generally have greater uranium concentration than sediments from other rock types (Rogers and Adams, 1970). Also, ground waters that circulate along fractures and faults may contribute significant amounts of uranium as well as other trace metals (Doi et al, 1975; Dyck, 1975). Consequently, the local geology may be one of the most important observations that will help in interpretation of the data.

<u>contamination</u> - All sources of contamination, such as mine waters, tailings, trash, and man-made structures (such as bridges, culverts, and well casings) are avoided where possible. However, any potential contaminant, such as uranium-rich phosphate fertilizers, is noted on the data form.

<u>vegetation</u> - In terrain containing abundant vegetation, relatively short dispersion trains in surface water can result. This generally results from organic matter absorbing uranium from the water and consequently increasing the uranium concentration in sediments (Dall'Aglio, 1971; Dyck et al, 1971).

FIELD MEASUREMENTS

Of the measurements commonly recorded in reconnaissance-scale exploration, the following are relatively easy to measure and are routinely taken by field personnel at all localities.

pH - In general, as pH decreases, uranium content increases. However, because uranium is soluble over such a wide range of pH (Grimbert and Loriod, 1968), its measurement is important to interpretation only when extreme values are encountered (Ostle and Ball, 1973).

conductivity - Uranium concentrations in waters of a given region generally correlate with concentrations of major components (approximated by conductivity), i.e., an increase in conductivity will usually correspond to an increase in uranium content in natural waters (MacDonald, 1969; Dall'Aglio, 1971; Dyck, 1975).

temperature - The temperature of water affects the rate of chemical and biological reactions which may influence the concentration of uranium (Fix, 1956; Ostle and Ball, 1973).

equivalent uranium - Scintillometer measurements of "shield in" (lead shield covers sensor) and "shield out" readings allow an equivalent uranium value to be calculated, which then can be used as a ground truth tie for airborne radiometric data. A high equivalent value may be an indication of mineralization of uranium and thorium daughter products (Whitehead and Brooks, 1969). However, scintillometer measurements are for total gamma radiation.

In general, field measurements in the Rocky Mountain states are taken by use of small, lightweight, battery-operated portable field instruments. Typical pH meters weigh about 0.5 kg, can be easily calibrated in the field, are temperature compensated, and have a ±0.1 pH precision. Spare probes can readily be exchanged. Conductivity meters, similar in size and weight to the pH meters, can measure up to 50 000 μmho/cm (±1%) and are easily calibrated by use of a standardized KCl solution.

All temperatures are measured with precalibrated thermometers. The air temperature in the shade is recorded to the nearest Celcius degree. The water temperature is usually recorded to the nearest 0.5°C. Ground radioactivity is measured with portable scintillometers.

In Alaska, instruments that combine pH, conductivity, temperature, and dissolved oxygen measurements are used. These water quality checkers are lightweight instruments having the versatility of making these measurements with only one piece of equipment, are battery operated, and can be recharged. These are also ideal for use in areas of difficult access. Temperature (0 to 40°C, ±0 5°C), conductivity (0-2000 μmho/cm, ±5 μmho/cm), pH (0 to 14, ±0.1 pH), and dissolved oxygen (0 to 20 parts per million (ppm), ±1.0 ppm) can be measured. Because of their versatility, these instruments are also being implemented for use in the Rocky Mountain states, and portable instruments mentioned above will be used as backup equipment.

SAMPLE COLLECTION

The LASL uses separate collection procedures for samples collected either in the Rocky Mountain states or in Alaska (Sharp and Aamodt, 1978).

Water samples. In the Rocky Mountain states, about 50 ml of water are collected in two 25-ml polyethylene vials that have been prewashed with dilute nitric acid. Ground-water samples are collected from either wells and springs as near the emergence source as possible. Holding tanks are not sampled. Stream waters are collected from the flowing current away from the bank. All waters are filtered through a 0.45-μ membrane and acidified to pH ≤ 1.0 with 8 M reagent-grade nitric acid. All water quality measurements are made with instruments previously discussed.

In Alaska, 50 ml of water are collected; however, due to the high purity of the water and the high transportation costs per sample location, the time-consuming operation of filtration is omitted. Field measurements include dissolved oxygen and are usually taken with water quality checkers, previously described.

Sediment samples. About 1 kg of sediment is collected from at least three adjacent spots at each location. The sediment must be water transported and taken below water level (if water is present) and must contain enough organic-rich, fine-grained particles to fill a 25-ml polyethylene vial. In lake areas in Alaska, the sediment is collected with a specially designed 11-kg, suction- operated bottom sampler that can be dropped from the side of a helicopter. The sample is usually collected with a polyethylene scoop and put into a rip-top polyethylene bag which is labeled both inside and out. The subcontractor dries the samples at $\leq 100°C$ and sieves the samples, retaining only the fraction that passes through a 100 mesh screen, which the LASL has determined is optimum for identification of uranium (Olsen, 1977).

Sampling densities. Based on extensive literature research of reconnaissance programs in similar terrains and based on the results of pilot studies (Sharp and Aamodt, 1976; Olsen, 1977), the LASL selected a nominal sample density of one location per 10 km^2 for the Rocky Mountain states. This sample density was selected to optimize recognition of uraniferous terranes, on a regional scale, for all physiographic provinces in LASL's sampling area. All sample locations in the Rocky Mountain states are preselected by LASL personnel. Surface streams are selected to represent drainage areas of about 10 km^2. Sites which cannot be reached in the field are reselected to approximate the original drainage area as closely as possible. For sampling in Alaska, a pattern containing 23-km^2 grids is marked on field maps, and helicopter pilots select all sample locations from lakes as near as possible to the center of each grid square. Alaskan streams are sampled at twice this density or two sample locations per 23 km^2.

SAMPLE AND DATA VERIFICATION

Occasionally LASL personnel collect control samples in the field. However, field supervision consists primarily of teaching samplers correct sampling procedures and checking to insure these procedures are followed.

After samples arrive at LASL, they are checked against the respective data form to make sure they are complete and properly labeled as described. Samples are sorted and then sent to the respective laboratories for analysis (Fig. 3).

After the data forms are keypunched, the data base is computer edited by a verification program to insure that all field data are consistent. Any samples that have inconsistent information, e.g., one sample with two sample numbers, are removed from the data base.

The LASL digitizes all sample locations from the field maps. The digitized results are generated by computer overlays to the field maps (usually 1:24 000 scale). Any locations plotted incorrectly are corrected and entered into the appropriate data base. Since a typical LASL contract area is 20 000 km^2 (with 2000 locations), more than 100 maps and overlays must be checked in this manner for each contract area.

ANALYTICAL PROGRAM

Ancillary Elements

In searching for uranium deposits, the major element of interest is obviously uranium. But certain other elements may form a much wider dispersion halo resulting from their chemical behavior and weathering characteristics and may act as supplemental indicators of uranium. The indicators or pathfinder elements most commonly used in uranium exploration are molybdenum, sulphur, lead, arsenic, vanadium, zinc, copper, nickel, and cobalt (Hawkes and Webb, 1962). Other elements, such as gold, tin, and tungsten can be analyzed for their own worth. Rare-earth elements provide a basis for in-depth geochemical studies, particularly with respect to the association of uranium with resistate minerals such as monazite. In general, the more elements sought, the more potential value the analytical data have. The particular elements selected for analysis depend on pilot surveys, analytical facilities, and funding constraints. In addition to the elements listed in Table I, LASL also reports molybdenum, arsenic, selenium, and zirconium (by x-ray fluorescence) in sediment, and vanadium (by plasma-source emission spectroscopy) and arsenic and selenium (by atomic absorption spectrometry) in water for certain areas, as requested by the DOE. The flow scheme for sample distribution is shown in Fig. 3.

Fluorometry Facility

All water samples are initially analyzed for uranium by fluorometry. Duplicate 0.20-ml water sample aliquots are transferred to special fabricated platinum dishes. Each set of samples is dried under heat lamps, then a 0.4 g pellet of 2% LiF-98% NaF flux is added to each dish and the samples are fused over rotary fusion burners. The fused pellets are transferred to one of four fluorometers, excited with ultra-violet radiation, and the fluorescence is recorded. The uranium concentrations are determined using a computer routine which compares the fluorescence from each pellet with those of pellets from standard uranium solutions and blanks run in the same set (Hues et al, 1977).

The lower limit of detection by normal procedures is 0.2 ppb. However, a sample that is found to have <0.2 ppb uranium is routinely reanalyzed after an evaporative concentration step that provides a 10X concentration factor. This step reduces the lower limit of detection of uranium in natural waters to 0.02 ppb. When a uranium concentration is <0.02 ppb, the sample is arbitrarily

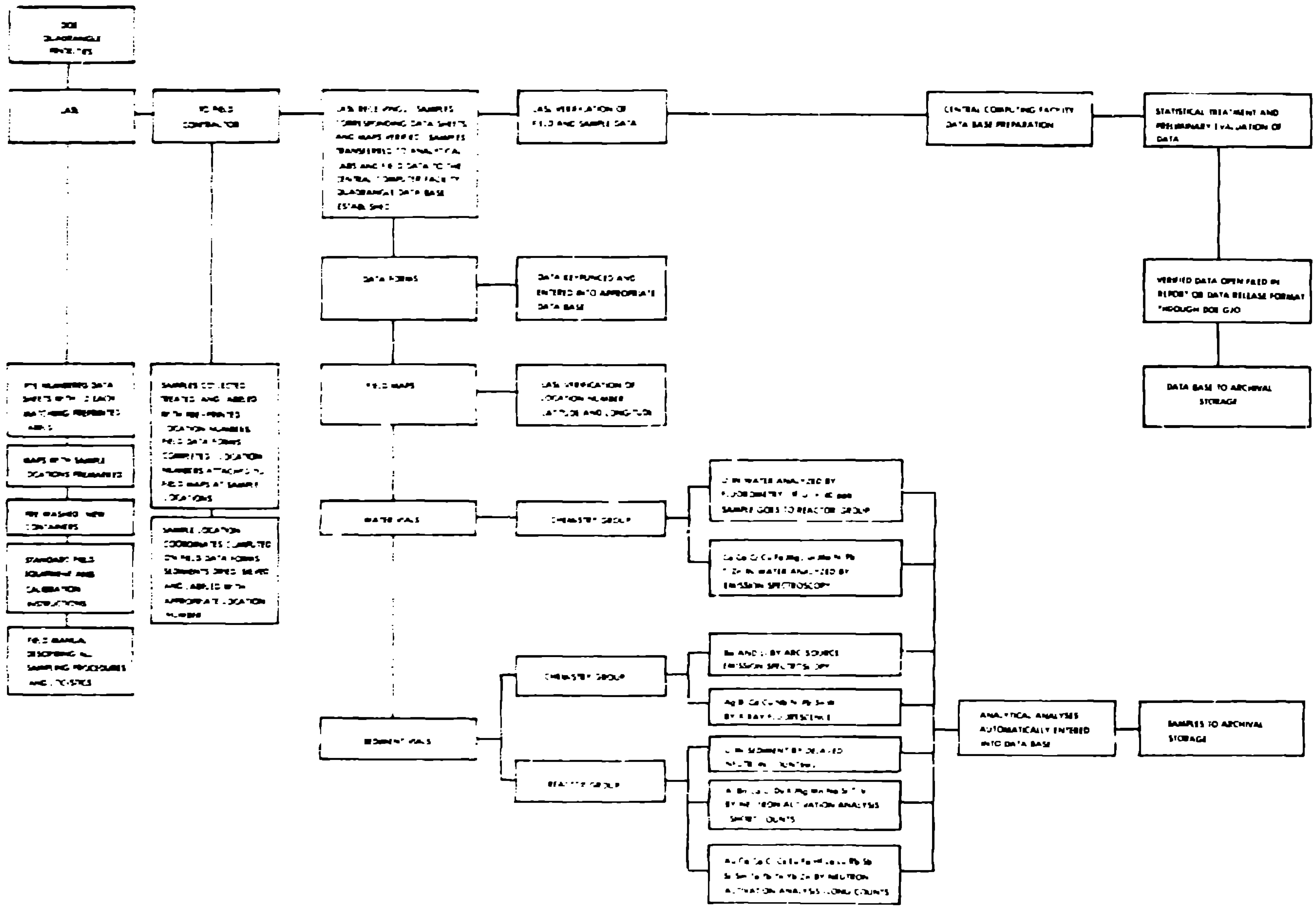


Fig. 3. Flow chart for the LASL HSSR program.

TABLE I

LIMITS OF DETECTION AND METHODS OF ANALYSES FOR MULTIELEMENT ANALYSES
(detection limits are ppm for sediment and ppb [illegible] water)

NEUTRON ACTIVATION ANALYSIS

Element	* Minimum Detection	Sample Medium
Al	200	Sediment
Au	0.1	Sediment
Ba	400	Sediment
Ca	5000	Sediment
Ce	10	Sediment
Cl	200	Sediment
Co	2	Sediment
Cr	20	Sediment
Cs	2	Sediment
Dy	2	Sediment
Eu	0.5	Sediment
Fe	2000	Sediment
Hf	1	Sediment
K	3000	Sediment
La	4	Sediment
Lu	0.2	Sediment
Mg	3000	Sediment
Mn	10	Sediment
Na	150	Sediment
Rb	25	Sediment
Sb	1	Sediment
Sc	0.1	Sediment
Sm	0.5	Sediment
Sr	300	Sediment
Ta	1	Sediment
Tb	1	Sediment
Th	0.8	Sediment
Ti	200	Sediment
V	5	Sediment
Yb	2	Sediment
Zn	30	Sediment

DELAYED NEUTRON COUNTING**

Element	Minimum Detection	Sample Medium
U	0.02	Sediment
U	0.2	Water

X-RAY FLUORESCENCE

Element	Minimum Detection	Sample Medium
Ag	[illegible]	Sediment
Bi	5	Sediment
Cu	10	Sediment
Nb	20	Sediment
Ni	15	Sediment
Pb	5	Sediment
Sn	10	Sediment
W	15	Sediment
Cd	5	Sediment

ARC-SOURCE EMISSION SPECTROSCOPY

Element	Minimum Detection	Sample Medium
Be	1	Sediment
Li	1	Sediment

PLASMA SOURCE EMISSION SPECTROSCOPY

Element	Minimum Detection	Sample Medium
Ca	20	Water
Co	55	Water
Cu	4	Water
Cr	25	Water
Fe	25	Water
Mg	2	Water
Mn	3	Water
Mo	25	Water
Ni	25	Water
Pb	200	Water
Ti	4	Water
Zn	50	Water

FLUOROMETRY**

Element	Minimum Detection	Sample Medium
U	0.02	Water

* Because of elemental interference, the detection limits for those elements determined by NAA will shift as a function of the composition of the sediment.

** All water samples having uranium concentrations >40 ppb are reanalyzed by delayed neutron counting.

assigned a value of 0.01 ppb. Samples having greater than 40 ppb are sent to the reactor group for reanalysis by delayed neutron counting (DNC).

Analyses are stored on a magnetic tape cassette and later transferred to the central computing facility. Throughput is presently 200 samples per day. The throughput for 1979 was about 40 000 analyses (Aamodt et al, 1979).

Energy Dispersive X-Ray Fluorescence

An energy-dispersive x-ray fluorescence system is used to determine nine elements in sediments (Table I). Two x-ray fluorescence analyzers have a throughput of 180-200 samples per day including blanks and standards. A third analyzer is used for special elements (such as molybdenum, arsenic, selenium, and zircon); however, if all three analyzers were used simultaneously 270-300 samples could be analyzed per working day (Morris et al, 1979).

Each system consists of an automatic 20-position sample changer, a lithium-drifted silicon detector, a pulsed molybdenum transmission-target x-ray tube, a multichannel analyzer, and a minicomputer. Sample splits are prepared for analysis by grinding 6 g of each sample to a minus 325-mesh powder. A computer program positions the 6-g samples in the x-ray beam, unfolds overlapping peaks, determines peak intensities for each element, and calculates the ratio of the intensity of each peak to that of the molybdenum K_α Compton peak. Concentrations of each element are then calculated using equations obtained by analyzing prepared standards. Detection limits are given in Table I. The relative standard deviation is 10% or less at the 100-ppm level and 20% or less at the 20-ppm level. Details of the method and equipment used are described by Hansel and Martell (1977).

Arc-Source Emission Spectrography

In sediments, two elements, beryllium and lithium, are analyzed by emission spectrography. A 7.5-mg portion of the minus 325-mesh sample that has already been analyzed by x-ray fluorescence is mixed with a graphite-silica buffer. This mixture is placed into a graphite electrode that is used as the anode of a dc arc. A 6-s exposure of the resulting spectrum is made on a direct-reading spectrograph. Photomultiplier tubes are used to measure the spectra lines of Be, Li, and V and the background spectra near these lines. The signals from the photomultiplier tubes are interfaced to a data acquisition system. Stabilization of the spectrograph is achieved by maintaining rigid temperature control of the grating and the focal curve containing the exit slits, and by providing two mercury reference lines between each sample analysis. Vanadium is monitored in order to correct for its interference with the beryllium determination. The results are simultaneously printed on paper and written on cassette tape for later transmission to a computer data file. The elemental concentrations of Be and Li are determined from the spectra, based on the results of previously run calibration standards. The lower detection limit for both elements is 1 ppm. Precision at the lower detection limit is ~50% for both and improves to ~25% at one order of magnitude above the lower limit. The throughput of this system is 200 analyses per day. In 1979, the throughput was more than 50 000 samples (Morris et al, 1979).

Plasma-Source Emission Spectrography

All water samples are now analyzed for 12 elements (Table I) by inductively coupled plasma emission spectroscopy. To allow complete system equilibration, the inductively coupled plasma and photomultiplier tubes are warmed up for at least 1 h prior to making any analyses. Argon coolant and sample carrier gas lines are adjusted and calibrated using a zinc standard. The sample solution is taken up from its container, nebulized, and injected into the plasma source at a rate of 9.2 x 10^{-9} m^3/s.

A plasma-therm inductively coupled plasma is used as the source. After the computer determines that the photomultiplier tubes have stabilized, a 15-s exposure of the resultant spectrum is made on a direct-reading spectrograph. Stabilization of the spectrograph is achieved by maintaining rigid control of the grating and exit slot focal curve temperatures, and by profiling two mercury reference lines between each sample analysis. Additional stability is attained by air-conditioning the room in which the spectrograph is situated. The resulting signals are read directly into a computer, and converted automatically to give the elemental concentrations. In addition, beryllium, sodium, and silicon are monitored. Interelement effects for each of the 15 elements monitored on the other elements are determined and used in correcting values for the 12 elements reported. Background corrections are made by running blanks, and control samples are run regularly. When high (off-scale) results are obtained, the computer calls for the insertion of a filter between the plasma source and the spectrograph, repeats the readings, and then converts and stores the corrected elemental concentrations. Analytical precision for the elements as determined for water by this method is ≈50% at the lower detection limit, improving to ≈10% one order of magnitude above the lower detection limit and to ≈5% two orders of magnitude above the detection limit.

The throughput of this system is 100 samples per 9-h day for samples exclusive of standards and controls. In 1979, the throughput was about 17 000 samples (Morris et al, 1979).

Certain elements analyzed by plasma-source emission spectrography are subject to certain analytical interferences. For example, large concentrations of calcium in a water sample may cause abnormal concentration readings for lead and molybdenum resulting in a lower precision of lead and molybdenum. Consequently, a dual-grating, high-resolution, direct reading spectrograph is under construction. This instrument will permit monitoring of 200 spectral lines and permit more accurate interelement-effect corrections.

Neutron-Activation Analysis

The neutron-activation analysis (NAA) system at the LASL represents a state-of-the-art system which produces precise values for uranium and 31 other elements in sediment samples. All samples are run through one of two computer-controlled pneumatic sample-handling systems. Each sample is irradiated twice and counted a total of three times (Table II): once for delayed neutron counting (DNC) for uranium and twice for gamma-ray spectrum counts for 31 other elements. Both sample transfer and data acquisition are entirely automated (Nunes and Weaver, 1978).

All sediment splits (regardless of which facility analyzes the sample) are made in a "clean room." Four-ml rabbits, the containers in which the sample undergoes irradiation, are loaded with approximately 5 g of sample, although as little as 0.5 g may be used. These rabbits are then loaded into a 50-sample transfer clip. The reactor pneumatic transfer system and background radiation levels are checked, and standards are run for calibration. The transfer clip is installed and the samples are cycled through the system.

The uranium concentration is automatically measured after 30 s by DNC, converted to ppm, and entered into the data base. The lower limit of detection of this method is 20 ppb (not ppm) uranium, which is below the range of uranium concentrations in natural sediment samples. Above the 1-ppm level,

TABLE II

STEPS INVOLVED IN IRRADIATION OF A SAMPLE
FOR NEUTRON ACTIVATION ANALYSIS

Steps	Operation	Elapsed Time (seconds)
1	20-s irradiation	20
2	10-s delay	30
3	20-s delayed neutron counting	50
4	20-min delay	1250
5	500-s γ-ray count (day shift) for Al, Ba, Ca, Cl, Dy, K, Mg, Mn, Na, Sr, Ti, V	1750
6	120-s irradiation	1820 (≈30 min)
7	2 week delay	2 weeks
8	900-s γ-ray count (night shift) for Au, Ce, Co, Cr, Cs, Eu, Fe, Hf, La, Lu, Rb, Sb, Sc, Sm, Ta, Tb, Th, Yb, Zn	

Note: There are two separate analytical systems. Each system has one delayed neutron counter and 4 Ge(Li) detectors and is capable of analyzing 200 samples per working day. A sample enters each system every 126 seconds.

the uranium values in sediment measured by DNC at the LASL have a one-sigma error of less than 4% (Aamodt et al, 1979). The specially designed delayed-neutron detectors, built by the LASL and used for these analyses, are described by Balestrini et al (1976).

After uranium is counted by DNC, 12 elements are determined by gamma counting. The remaining 19 elements are determined by γ-ray counting following the re-irradiation and two week delay. The γ-ray counting is done by lead-shielded Ge(Li) detectors; the 4096-channel γ-ray data are recorded and subsequently analyzed for each individual element by computer. The analytical data for each sample are automatically printed out along with the associated statistical errors. The data are stored on magnetic disk tapes until they can be transferred to the central computing facility and entered into the appropriate HSSR data base. Current "typical" lower detection limits for the elements determined by NAA are in the ppm range as reported in Nunes and Weaver (1978); however, the actual detection limit for an element depends upon the composition of the sample, so this limit may be higher or lower than the "typical" value. At concentration values one order of magnitude above the lower detection limits, the relative errors are generally less than 10%.

Both systems have a combined total throughput of 400 samples per day. The yearly throughput greatly exceeds 50 000 samples.

Uranium Determination in Water Samples by DNC

Only waters with >40 ppb uranium (as determined by fluorometry) or those with impurities that cause interference with uranium-induced fluorescence are analyzed using DNC. Samples are transferred to clean rabbits before being analyzed. Each water sample is weighed, then loaded into a 25-sample transfer clip. The reactor pneumatic transfer system and background radiation levels are checked and four standards are run for calibration. The transfer clip is installed on the pneumatic feed line and the samples are cycled through the system. Typically, a 60-s irradiaton, 30-s delay, and 60-s count cycle is used. The neutron count rate is automatically measured, converted to ppb uranium, and entered into a computer data base. The lower detection limit for uranium in water by DNC as used at the LASL is 0.2 ppb. The statistical error of this method is ≈20% at a uranium concentration of 1 ppb, ≈6% at 10 ppb, and <4% at 40 ppb or greater. Statistical treatments of uranium concentrations obtained from the same suites of samples analyzed both by fluorometry and DNC have shown that there is no significant difference between the results of the two analytical methods as used at the LASL. This analytical comparability is rechecked periodically.

Central Computer Facility

The LASL data-base management group employs a general-purpose, data-base management system (Nie et al, 1975) to maintain and organize the field and analytical data for the HSSR project. The data-base management system is used with about 75 programs to load the field data, check the data for consistency, and load analytical results (Cheadle, 1977 and 1978).

The samples and analytical data are grouped into data bases according to NTMS quadrangle boundaries (Sharp, 1977). Each data base stores about 138 variables for each sample and occupies about 125K words of core. At present, there are about 220 000 sample locations stored in 158 data bases; some data bases are in the process of being loaded, some are in mass storage, and some are on disk. Each sample location is associated with a number of coded field measurements and observations and up to 13 water and 43 sediment analyses. There are about 25 million pieces of information in readily available form (C. McInteer, personal communication, LASL, 1980). A double tape backup on each data base is maintained and updated after each loading operation.

EVALUATION OF DATA

Once all samples from an area have been analyzed for uranium and other elements, the measured concentrations are entered into the appropriate data base at the central computing facility (Fig. 3). A maximum effort is made by all LASL personnel to check the data to make sure they are analytically correct before being released.

Each report contains a suite of standard histograms for each sample type (stream waters, stream sediments, etc.). These histograms are used both to evaluate the data and to establish reasonable limits in selecting intervals for the various concentration overlays, which are also included with each report. These concentration overlays are all produced at a 1:250 000 scale to be used in conjunction with the appropriate 1:250 000-scale geologic and topographic maps. Geologic maps, when not available at this scale, are compiled and included with the reports. Concentration overlays, one each for a sample location, a uranium concentration in waters, conductivities in waters, uranium concentrations in sediments, and thorium concentrations in sediments, are included with the report. An example of a uranium concentrations overlay for sediment is shown in Fig. 4. Appendixes include listings of field data and

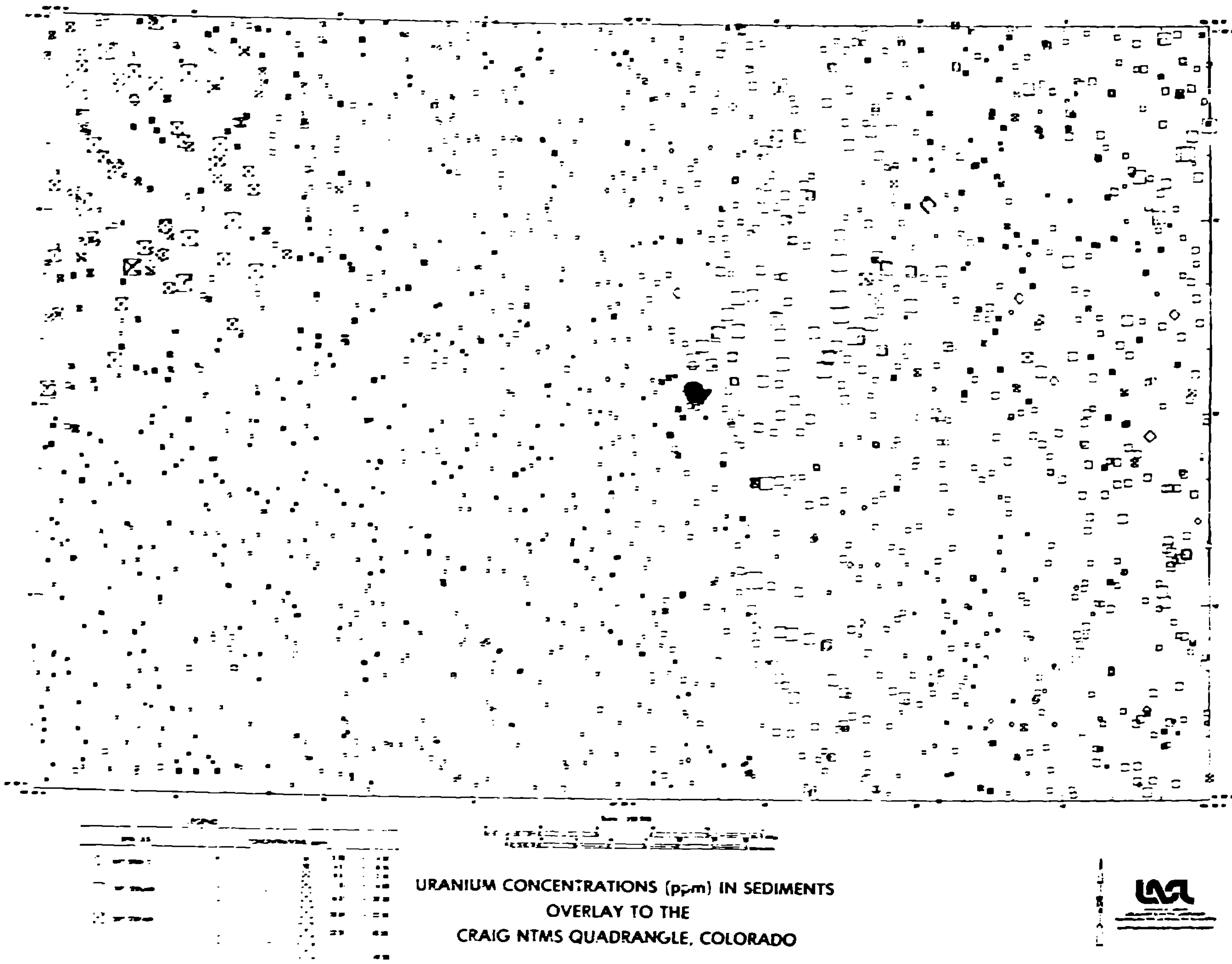


Fig. 4. A reduction of a uranium concentrations in sediments overlay (plate IV in Bolivar and Hill, 1979.)

elemental concentrations for both waters and sediments and a summary of standard LASL HSSR procedures and codes.

Standard reports also contain approximately 10 to 20 pages of text that includes sections on geography, weather, geology, hydrology, geochemical considerations, and known uranium occurrences in the area. Empirical and statistical evaluations are concise and generally refer to uranium data only; however, all analytical data are included in every report. Data releases are an abbreviated type of report and include only histograms, overlays, and data listings. All reports are made available to the public in both magnetic tape or report form.

Potential Uses of Data

The LASL personnel are now examining the analytical data in great detail and have been employing several multivariate statistical programs as an aid in their evaluation. For example, Bolivar and Hill (1979), in an HSSR report of the Craig NTMS quadrangle, Colorado, were able to delineate several areas favorable for uranium mineralization using empirical evaluation methods. Planner (1980) showed a correlation between structure and uranium concentrations in this quadrangle by employing a "moving average" method of data evaluation. Also, by using both a moving average and krigeage techniques, he was able to identify a known area of mineralization that the evaluation of Bolivar and Hill (1979) did not substantiate.

By use of matrix correlation coefficients, scattergram plots, and factor analysis, Beyth et al (1980) were further able to identify and pinpoint areas in the Craig quadrangle favorable for uranium and base metal mineralization. Some areas have characteristic low uranium concentrations in waters and sediments and general examination of data from these areas may not reveal potential exploration regions. However, sophisticated statistical treatment of the data helps to identify these areas. Furthermore, these latter studies dramatically reveal some of the potential uses of the multielement data in an HSSR report.

Other potential uses of HSSR data include trace-element characterization of mineral deposits, strategic metals evaluations, water quality studies, a ground truth data set for remote sensing, academic studies involving the relation of elements to geologic formations, health studies, establishing base lines for pollution studies and compiling a national geochemical atlas. The industrial and scientific communities are only beginning to utilize and realize the potential of the enormous volume of data generated by the HSSR.

SUMMARY

Since April 1975, the LASL has established an effective and capable hydrogeochemical and stream sediment reconnaissance program. Based on an extensive review of world literature (Sharp and Bolivar, 1980), the results of several pilot studies (Sharp and Aamodt, 1976; Olsen, 1977; Aamodt, 1978; Sharp et al, 1979), and in keeping within funding constraints, the LASL has developed standardized sampling procedures (Sharp and Aamodt, 1978) that emphasize the determination of uranium concentrations in both natural waters and waterborne sediments. Water and/or sediment samples are collected from approximately each nominal 10 km^2, except in lake areas of Alaska where samples are collected every nominal 23 km^2. The LASL area includes the states of New Mexico, Colorado, Montana, Wyoming, and Alaska in which 165 000 waters and 185 000 sediments have been collected (as of May 1980) from 250 000 locations (Bolivar, 1979).

In order to implement this program, LASL has developed automated, precise, and comprehensive analytical systems with extremely large throughput capabilities. For example, over 50 000 samples are analyzed for 31 elements annually by neutron activation. Complementary statistical routines were developed to manage the tremendous quantity of analytical data generated in this study (Cheadle, 1977 and 1978; Kosiewicz, 1979).

All water samples are analyzed for uranium by fluorometry (Hues et al, 1977) and for 12 other elements (Table I) by inductively coupled plasma emission spectroscopy.

All sediment samples are analyzed for uranium by delayed neutron counting and for 31 other elements (Table I) by neutron activation analysis. Sample transfer and data acquisition are entirely automated and represent a state-of-the-art system (Nunes and Weaver, 1978). An additional 11 elements are determined in sediment samples by energy dispersive x-ray fluorescence (Hansel and Martell, 1977) and arc-source emission spectroscopy.

Data are reported according to NTMS quadrangle boundaries. Data are released to the public in both printed copies and magnetic tapes. Each report contains data listings for each sample location; each location has a number of field measurements and observations and may have as many as 13 element analyses in water and 43 element analyses in sediment samples. Histograms for each sample type and concentration overlays for sample locations, uranium concentrations in water, uranium concentrations in sediments, conductivities in waters, and thorium concentrations in sediments are also included. All overlays are at a 1:250 000 scale for use with NTMS topographic and geologic maps.

Refined statistical techniques are now being used to identify geochemically anomalous areas, to recognize base-metal regions, and to aid in identifying mineral associations (Beyth et al, 1980; Planner, 1980). Such studies greatly emphasize some of the potential uses of HSSR data. Other potential uses include trace-element characterization of mineral deposits, water quality studies, resource characterizations, and compilation of a national geochemical atlas.

The LASL HSSR program has been developed to aid in a national uranium resource evaluation. Because many elements are analyzed in addition to uranium and because the LASL program employs standard geochemical sampling techniques and analytical methodology, this program technology could be adopted for regional studies of economic commodities in almost any country.

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